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METHODS OF CHEMICAL ANALYSIS OF SOFT SOLDERS FOR JOINTING ALUMINIUM AND ALUMINIUM ALLOYS

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF SOFT SOLDERS FOR JOINTING ALUMINIUM AND ALUMINIUM ALLOYS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 23 June 1976, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 In order to determine correctly the composition of different grades of soft solders for jointing aluminium and aluminium alloys prescribed in IS : 5479-1969*, standard methods of analysis have been laid down in this standard.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and the practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been achieved by deriving assistance from ' 1973 Book of ASTM Methods for chemical analysis of metals, Part 32 ', issued by the American Society for Testing and Materials.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

1. SCOPE

1.1 This standard covers the methods of chemical analysis of tin, lead, zinc, antimony, arsenic, copper, iron and bismuth in soft solders for jointing aluminium and aluminium alloys as specified in IS : 5479-1969*.

2. QUALITY OF REAGENTS

2.1 Unless otherwise specified, pure chemicals and distilled water (*see* IS : 1070-1960‡) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Specification for solders for jointing aluminium and aluminium alloys.

†Rules for rounding off numerical values (*revised*).

‡Specification for water, distilled quality (*revised*).

3. DETERMINATION OF TIN BY THE IODOMETRIC METHOD

3.1 Outline of the Method — Tin is reduced in hydrochloric acid medium by pure aluminium. Reduced tin is titrated with standard potassium iodate solution using starch as indicator.

3.2 Reagents

3.2.1 *Concentrated Sulphuric Acid* — rd = 1·84 (conforming to IS : 266-1961*).

3.2.2 *Potassium Bisulphate* — solid.

3.2.3 *Antimony Trichloride Solution* — 1 percent in hydrochloric acid.

3.2.4 *Concentrated Hydrochloric Acid* — rd = 1·16 (conforming to IS : 265-1962†).

3.2.5 *Pure Aluminium* — foil or drillings.

3.2.6 *Sodium Bicarbonate Solution* — saturated.

3.2.7 *Potassium Iodide Solution* — 5 percent (*m/v*).

3.2.8 *Starch Solution (10 g/l)* — Make a paste of 1 g of soluble starch in about 5 ml of water and add to 100 ml of boiling water.

3.2.9 *Standard Potassium Iodate Solution (0·1 N)* — Dissolve 3·567 g of potassium iodate, previously dried at 160 to 180°C to constant mass, in 200 ml of water. Add 1 g of sodium hydroxide and 10 g of potassium iodide. Transfer the solution to a 1 000-ml volumetric flask, dilute with water to the mark and mix.

3.3 Procedure

3.3.1 Weigh accurately about 0·4 g of the sample preferably containing 0·10 to 0·30 g of tin to a 500-ml flask. Add 20 ml of concentrated sulphuric acid and 5 g of potassium bisulphate and heat to decompose the sample. Avoid too high temperatures during the initial heating. When decomposition is complete, heat vigorously over an open flame to expel sulphur completely. Cool, add 180 ml of water.

3.3.2 Add 2 to 3 drops of antimony trichloride solution. To the solution add 60 ml of concentrated hydrochloric acid and about 4 g of pure aluminium, add little at a time. Connect the flask (*see* Fig. 1). Heat to boiling and boil with continuous evolution of gas for at least 30 minutes in order to dissolve aluminium completely.

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*Specification for sulphuric acid (*revised*).

†Specification for hydrochloric acid (*revised*).

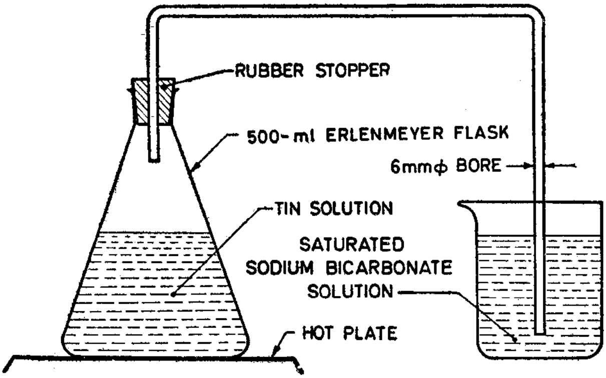

FIG. 1 APPARATUS FOR REDUCTION OF TIN

3.3.3 After reduction is complete, cool the contents of the flask to about 10°C, maintaining an atmosphere of carbon dioxide (CO_2) by dipping the end of outlet tube into a saturated solution of sodium bicarbonate. Open the flask, add 5 ml of potassium iodide solution and 5 to 10 drops of starch solution. Titrate with standard potassium iodate solution to permanent blue tint.

3.4 Calculation

$$\text{Tin, percent} = \frac{A \times B}{C} \times 100$$

where

A = volume in ml of standard potassium iodate solution required to titrate the sample,

B = tin equivalent of the standard potassium iodate solution in g/ml, and

C = mass in g of the sample used.

4. DETERMINATION OF LEAD BY LEAD SULPHATE (GRAVIMETRIC) METHOD

4.1 Outline of the Method — After separation of tin and antimony as volatile bromide, lead is precipitated as lead sulphate. The precipitate is washed, ignited and weighed.

4.2 Reagents

4.2.1 *Hydrobromic Acid-Bromine Mixture* — Add 10 ml of bromine in 90 ml of hydrobromic acid.

4.2.2 *Perchloric Acid* — 70 percent.

4.2.3 *Hydrobromic Acid* — 48 percent.

4.2.4 *Dilute Sulphuric Acid* — 1 : 1 (*v*/*v*).

4.2.5 *Lead Acid Solution* — Dissolve 0·5 g of lead nitrate in 200 ml of water and add with stirring 5 ml of concentrated sulphuric acid (rd = 1·84) to the solution. Allow to stand for 24 hours and siphon or decant through a filter pulp. Discard the precipitate.

4.3 Procedure

4.3.1 Transfer 0·2 g of the sample to 250-ml of wide-mouthed flask. Add 20 ml of hydrobromic acid-bromine mixture. Gently heat till the dissolution is complete, avoiding excessive loss of bromine. Add 5 ml of perchloric acid and heat over an open flame until white fumes first appear. Then heat moderately to decompose lead bromide and to expel all hydrobromic acid, avoiding excessive loss of perchloric acid.

4.3.2 If the solution is turbid, add 5 ml of hydrobromic acid and again heat to expel hydrobromic acid and to decompose lead bromide. Repeat the process till the solution is clear. Then add 10 ml of dilute sulphuric acid and evaporate to dense white fumes to expel all perchloric acid. Continue heating until the volume of the solution is reduced to about 5 ml.

4.3.3 Cool, add 100 ml of lead acid solution and boil gently to dissolve the soluble salts. Cool to 50°C and allow to stand for 1 to 2 hours. Cool to room temperature. Filter through sintered Gooch crucible No. 4 and wash the precipitate well with lead acid solution. Reserve the filtrate for zinc determination. Dry the crucible at 110-120°C to constant mass.

4.4 Calculation

$$\text{Lead, percent} = \frac{A \times 0{\cdot}682\,2}{B} \times 100$$

where

A = mass in g of lead sulphate, and

B = mass in g of sample used.

5. DETERMINATION OF ZINC BY EDTA (VOLUMETRIC) METHOD

5.1 Outline of the Method — After removing all interfering elements, zinc is precipitated as zinc sulphide in ammonium formate-tartaric acid medium. The precipitate is dissolved in hydrochloric acid and determined volumetrically by titrating with EDTA solution.

5.2 Reagents

5.2.1 *Hydrogen Sulphide* — gas.

5.2.2 *Dilute Sulphuric Acid* — 1 percent, saturated with hydrogen sulphide.

5.2.3 *Tartaric Acid Solution* — 35 percent (*m/v*).

5.2.4 *Formate Mixture* — Add 500 g of ammonium sulphate in 400 ml of formic acid (rd = 1·2), and 60 ml of ammonium hydroxide. Make up to 2 000 ml with water.

5.2.5 *Formate Wash Solution* — Dilute 25 ml of the solution (*see* **5.2.4**) to 1 000 ml with water and saturate with hydrogen sulphide.

5.2.6 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

5.2.7 *Methyl Red Indicator Solution* — 0·1 percent (*m/v*).

5.2.8 *Sodium Hydroxide Solution* — 200 g/l (*m/v*).

5.2.9 *Potassium Cyanide Solution* — 100 g/l. Dissolve 100 g of potassium cyanide in 500 ml of water; dilute to one litre.

5.2.10 *Buffer Solution* (*10* p*H*) — Dissolve 54 g of ammonium chloride (NH_4Cl) in 300 ml of water, add 300 ml of ammonia (rd = 0·91) and dilute to one litre.

5.2.11 *Eriochrome Black-T Indicator Solution* — Dissolve 0·4 g of the indicator in 20 ml of ethanol, add 30 ml of triethanolamine and store in a polyethylene dropping bottle.

5.2.12 *Formaldehyde* (1 : 9) — Dilute 100 ml of formaldehyde (37 percent) with 900 ml of water.

5.2.13 *Standard EDTA Solution* (*0·01 M*) — Dissolve about 3·728 g of the disodium salt of EDTA $[(OOC.CH_2)_2NH.CH_2CH_2\ NH\ (CH_2\ COO)_2 Na_2 .\ 2H_2O]$ in water and transfer to a 1 000 ml volumetric flask, dilute to the mark and mix.

5.2.14 *Hydrobromic Acid-Bromine Mixture* — Mix 10 ml of bromine with 90 ml of hydrobromic acid.

5.2.15 *Perchloric Acid* — 70 percent.

5.3 Procedure for Solders Containing Tin, Antimony and Lead

5.3.1 Take the filtrate reserved under **4.3.3** and heat to 50°C and saturate with hydrogen sulphide gas. Filter through paper pulp pad to remove copper, tin, and antimony if present. Wash with dilute sulphuric acid. Boil the filtrate until free from hydrogen sulphide gas and dilute to about 200 ml.

5.3.2 Add 25 ml of tartaric acid solution and make ammoniacal to methyl red. Add 0·5 ml in excess. Add 25 ml of formate mixture and dilute to 400 ml. Bring nearly to boiling and pass hydrogen sulphide gas for 15 minutes. Allow to stand for 30 minutes at a temperature of 60-70°C, then for 90 minutes at room temperature. Filter through ashless paper pulp pad washing 5 to 6 times with formate wash solution.

5.3.3 Transfer the paper along with the precipitate into the original beaker and add 20 ml of dilute hydrochloric acid. Heat to dissolve the precipitate, dilute to about 100 ml and boil to expel hydorgen sulphide gas. Cool, filter and wash the paper thoroughly with hot water till free from acid. Transfer the solution to an 800-ml beaker.

5.3.4 Add 3 to 4 drops of methyl red indicator and carefully neutralize the solution with sodium hydroxide solution. Add 10 ml of potassium cyanide and 150 ml of buffer solutions and dilute to about 600 ml with water. Add 5 drops of eriochrome black-T indicator and sufficient formaldehyde to just give a red colour. Titrate slowly with EDTA solution to a bluish green end point. Add 10 ml of formaldehyde and, if the colour changes to red, titrate again with EDTA solution to bluish green end point. Continue with the formaldehyde addition and EDTA titration till the addition of formaldehyde has no effect within 2 minutes on the end point.

5.4 Calculation

$$\text{Zinc, percent} = \frac{A \times B}{C} \times 100$$

where

A = millilitres of EDTA required for titrating the solution,
B = EDTA equivalent in terms of g of zinc per ml, and
C = mass in g of the sample taken.

5.5 Procedure for Solders Containing Tin and Antimony But no Lead

5.5.1 Transfer 0·5 g of an accurately weighed sample in a 250-ml wide-mouthed flask and add 20 ml of hydrobromic acid-bromine mixture. Gently heat till the dissolution is complete avoiding excessive loss of bromine. Add 5 ml of perchloric acid and heat over an open flame in a fume cupboard until white fumes first appear, then heat moderately for 5-10 minutes avoiding excessive loss of perchloric acid.

5.5.2 Cool the solution, dilute with water to 250 ml in a volumetric flask. Take an aliquot containing about 10-20 mg of zinc and continue as in **5.3.1** to **5.4**.

6. METHOD FOR DETERMINATION OF ANTIMONY BY THE POTASSIUM BROMATE METHOD

6.1 Follow the procedure as given in IS : 1940-1969*.

7. METHOD FOR DETERMINATION OF ARSENIC BY THE MOLYBDENUM BLUE (PHOTOMETRIC) METHOD

7.1 Follow the procedure as given in IS : 1940-1969*.

8. METHOD FOR DETERMINATION OF COPPER BY THE CUPRIC BROMIDE (PHOTOMETRIC) METHOD

8.1 Take 0·4 to 1·0 g of sample and proceed as in IS : 1940-1969*.

9. METHOD FOR DETERMINATION OF IRON BY THE ORTHO-PHENANTHROLINE (PHOTOMETRIC) METHOD

9.1 Follow the procedure as given in IS:1940-1969*.

10. METHOD FOR DETERMINATION OF BISMUTH BY THE THIOUREA (PHOTOMETRIC) METHOD

10.1 Take 0·5 to 1·0 g of sample and proceed as in IS : 1940-1969*.

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*Methods of chemical analysis of tin ingot (*first revision*).

INDIAN STANDARDS
ON
METHODS OF CHEMICAL AND SPECTROGRAPHIC ANALYSIS

IS:

Aluminium

504-1963	Aluminium and its alloys (*revised*)

Copper and Copper Alloys

440-1964	Copper (*revised*)
3186-1965	Cadmium copper
3187-1965	Copper-nickel-zinc alloys
3685-1966	Brasses
3883-1966	Copper-tellurium alloys
4027-1967	Bronzes
6517 (Part I)-1972	Copper master alloys: Part I Copper-nickel alloy

Spectrographic Analysis

2271-1967	Platinum
2599-1963	High purity zinc and zinc base alloys for die casting
6010-1971	Recommended practice for photographic processing in spectrochemical analysis

Lead, Zinc, Tin, Antimony and Their Alloys

403-1964	Lead and antimonial lead (*revised*)
406-1964	Slab zinc (spelter)
1047-1965	Antimony (*revised*)
1345-1960	Printing metals
1409-1959	Antifriction bearing alloys
1940-1969	Tin ingot (*first revision*)
2600-1964	High purity zinc and zinc base alloys for die casting
6516-1972	Tin and lead in secondary tin

Nickel

1952-1963	Nickel anodes
2390-1967	Foundry nickel
2766 (Part I)-1968	Primary nickel: Part I

Refractories

1335-1959	Alumina in refractory materials (*tentative*)
1527-1972	High silica refractory materials (*second revision*)

Solders and Brazing Alloys

964-1956	Silver solder
998-1959	Soft solder
999-1959	Brazing solder
4354 (Part I)-1967	Magnesium-aluminium brazing alloys: Part I Aluminium, manganese, zinc and silicon
4548 (Part I)-1967	Copper-gold brazing alloys: Part I Gold and copper
4646 (Part I)-1968	Chemical analysis of copper-phosphorus brazing alloys: Part I Silver and copper
4667 (Part I)-1968	Silver-copper brazing alloys: Part I Silver and copper
4667 (Part II)-1969	Silver-copper brazing alloys: Part II Silver, copper and tin
4703-1968	Silver-manganese brazing alloys

General

1338-1958	Certified samples for metallurgical analysis